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DRY-PROCESSING OF LONG-TERM WET-STORED FLY ASH FOR USE AS AN ADDITION IN CONCRETE

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Dry-processing of Long-term Wet-stored Fly Ash for Use as an Addition in Concrete

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Abstract
The paper describes a study carried out to explore the potential for recovery of fly ash from wet-storage areas for use as an addition in concrete, and involved drying and processing material to achieve finer fractions/reduce particle size. Three relatively coarse fly ashes with medium/high carbon contents (loss-on-ignition; LOI) were obtained from two UK lagoons and a stockpile. Initially, drying (105 ºC) and screening (600 µm) were carried out, giving acceptable handling properties. Thereafter, processing using (63 µm) sieving, air classifying or grinding was investigated. The methods gave increased fineness levels (greatest with grinding), with some reductions in LOI also found after sieving and air classifying. Assessment of fly ash reactivity indicated improvements with processing, and most noticeable effects by grinding. Tests on concrete found that both consistence (slump) and compressive (cube) strength increased with processing and tended to follow fly ash fineness. Further analysis showed that strength could be related to the sub 10 µm particle level in fly ash. This appeared to be irrespective of processing method, with progressive increases (in strength) up to sub 10 µm contents of about 60% (maximum in the study). Concrete tests for water absorption and intrinsic (air) permeability, carbonation and chloride diffusion showed that these were also influenced by fly ash sub 10 µm content. The factors affecting behaviour are discussed and practical implications of the research considered.

1. Introduction

Fly ash has found increasing use as an addition (cement component) in recent years, giving benefits to concrete's performance, including aspects of durability, and environmental impact [1, 2]. With moves from coal-firing in electricity generation to alternative methods, the availability of fly ash for concrete is being affected [3]. As a result, changes to sourcing material are likely, with probable developments in seaborne trade [4]. Furthermore, attention is being given to the potential for using the reserves of wet-stored fly ash (from lagoons and stockpiles) that have been collected around power stations [5], in some cases, over many years. For example, in the UK, quantities of about 50 Mt of fly ash are believed to be available for recovery [6].

Studies on siliceous fly ash (Class F, ASTM C618 [7]) in lagoons and stockpiles suggest material may be variable, with a range of fineness levels and moderate to high carbon contents [8]. Short-term tests on wet-stored fly ash (laboratory and site, mainly up to 6 or 18 months) indicate that agglomeration occurs, with minor changes in bulk chemistry, and the presence of sulfate-based products on particle surfaces/reduced free lime contents also noted [9, 10]. These were influenced by fly ash properties (e.g. free lime content), storage conditions (e.g. moisture level) and holding period [10], with reactivity potential affected [9, 11]. Wet storage effects at actual fly ash sites (various exposure conditions/periods) have been noted to range from little change in the material [12, 13], to processes including dissolution of components at particle surfaces/formation of secondary phases [13-15]. Hence, evaluation of individual storage areas is likely to be a necessary part of fly ash recovery.

Research indicates that one route to using wet-stored siliceous fly ash as an addition in concrete could be through adjustments to the mix, e.g. superplasticizing admixture dose and w/c ratio, to match the fresh and hardened properties of concrete containing dry material [16]. However, a practical difficulty with wet-stored fly ash, that may be encountered in concrete production, is handling and transportation. Indeed, this appears to be reflected in limits on moisture level, or reference to drying of fly ash (before use), covered in Standards for the material [7, 17]. Laboratory tests on wet fly ash in a small aggregate hopper, suggest that issues including the quantity of material, the period it is stored for (before discharge) and vibration or forced action at the hopper outlet are important with regard to use in wet form [12].

Another option for using lagoon or stockpile fly ash in concrete, could then be through drying and processing. Drying of fly ash can enable recovery of handling properties following wet storage and has been used with the material previously on a large-scale [18]. Thereafter, techniques for dry processing of fly ash could be used, if required, to achieve properties for concrete (e.g. to BS EN 450-1 [17]), or else to potentially enhance performance. This paper reports on a study carried out to investigate the feasibility of this approach to recover wet-stored siliceous fly ash for use as an addition in concrete. Note that fly ash with higher lime contents (Class C to ASTM C618 [7]) is likely to be more affected by wet-storage (c.f. self-setting) and this type of material was not, therefore, considered in the work described.

2. Research Programme

Wet fly ash samples were obtained from three UK power stations (two from lagoons and the other a stockpile). These were initially dried (at 105 ^{o}C) and passed through a screen (600 μm) before processing, which was carried out either by (i) separating particles (63 μm sieving or air classifying), or (ii) directly reducing their size (grinding). These techniques were selected as there is experience using them with dry fly ash, at full or laboratory-scales, and they are familiar to the fly ash/cement industries. Given the carbon levels (loss-on-ignition (LOI)) of the fly ashes (with two around the BS EN 450-1 [17] limit (9.0%)) no attempt was made, at this stage, to directly achieve reductions by processing.

The materials were physically and chemically characterized before and after processing to evaluate the effects occurring. Tests were also made to consider the influence of processing on hydration/pozzolanic activity of the PC/fly ash combinations, with heat development and calcium hydroxide ($Ca(OH)_2$) levels evaluated in

paste. The study then examined the materials in concrete of average structural strength class. This included initial tests on fresh properties (consistence (slump)) and compressive (cube) strength development, before considering water absorption and intrinsic (air) permeability, and aspects of durability (carbonation and chloride diffusion).

3. Materials

3.1 Sources and Properties of Recovered Fly Ashes

Recovered fly ash was sourced from three storage areas, with two from lagoons (LFA1 and LFA2; bituminous coal) and the other from a stockpile (SFA1; anthracitic coal). Given the storage periods involved, and changes in coal/burning conditions with time, it was not possible to obtain reliable reference materials. Comparisons were, therefore, made with regard to Standard requirements (BS EN 450-1 [17]), which is likely to be the approach that would be followed in a practical recovery situation. A summary of the test methods [19-21] used to investigate the fly ash characteristics is given in Table 1.

LFA1 was from a lagoon where material had been deposited over the previous 30 years. LFA2 had been introduced to its lagoon about 3 years prior to sampling. SFA1 was kept in a stockpile at the site for 4 to 5 years. The lagoons had been drained at the time of recovery, enabling access for this to be carried out by hand auger. Samples were taken at a small distance from exposed fly ash surfaces. The typical moisture contents of LFA1 and 2 on receipt at the laboratory were 43.8 and 19.3% respectively, while that of SFA1 was 27.5%.

The physical and chemical characteristics of recovered fly ashes (following oven drying at 105 ^{o}C) are given in Table 2. LFA1 and LFA2 had fineness levels above the 40.0% limit (retained on a 45 μm sieve) of BS EN 450-1 [17], while SFA1 was slightly below this, with a similar ranking noted between these and particle size distribution data. Such properties are, in part, likely to reflect wet-storage (agglomeration) effects referred to above. The LOI of LFA1 was 6.4%, while LFA2 and SFA1 were 10.3% and 15.1%, with both, therefore, exceeding the BS EN 450-1 [17] limit. Earlier work [9, 10] suggests increases in LOI of around 0.5 to 1.0% can be obtained after wet-storage (6 months, laboratory) compared to dry material, reflecting decomposition (during the test) of reaction products formed. The particle densities are around what may be expected for the materials, given the above properties, and can also be influenced by wet storage, as noted previously.

The sum of the main oxides (silica (SiO_2) + alumina (Al_2O_3) + iron oxide (Fe_2O_3)) was greater than 70%, with calcium oxide (CaO) contents less than 3.0%, alkalis (Na_2O_{eq}) 1.7 to 2.1% and sulfate (SO_3) 0.5 to 0.6%. The latter three are at the lower end of the typical range [22] and may relate to the loss of soluble components from particle surfaces during wet storage [9]. Typically potassium oxide (K_2O) is higher in fly ash than sodium oxide (Na_2O) [23] and the differences with LFA1 may reflect the presence of (sodium) chloride (Cl) in the material (also noted in LFA2). This is likely to reflect salt water used to wet fly ash, or the site location (estuarine/coastal), and would be an issue for steel in concrete. The crystalline contents were lower/non-crystalline higher in SFA1 than LFA1 and LFA2, which had similar values.

3.2. Other Materials

A single Portland cement (PC, CEM1, Strength Class 42.5N) to BS EN 197-1 [24] was used in the reactivity and concrete tests and its main characteristics are given in Table 2. The aggregates, to BS EN 12620 [25], were natural gravel in 10/20 and 4/10 mm sizes and a medium grade sand. The particle densities for coarse and fine aggregate were 2620 and 2600 kg/m^3 with water absorption 0.8 and 0.9% (laboratory dry to saturated surface dry). The aggregates were laboratory dried before use.

4. Preparation of Specimens and Test Methods

Paste specimens (w/c ratio, 0.5; 30% fly ash in cement) for PC/fly ash reactivity studies were prepared by hand mixing. Concrete (w/c ratio 0.53; 30% fly ash in cement) was produced in a horizontal, forced action, laboratory pan mixer following the method described in BS 1881, Part 125 [26]. Heat development monitoring (of paste) was initiated following mixing. For the other tests, paste/concrete, the cast speciments were seal-cured/stored under damp hessian and polythene sheeting in the laboratory for the first 24 hours, with curing in water at 20 ^oC, thereafter. A summary of the test methods, essentially following procedures described in British/European standards, or techniques developed at the University of Dundee [27-32] are given in Table 3, with curing carried out until pre-treatment/testing at 28 days, unless noted otherwise.

5. Processing Techniques

Given the moisture contents and characteristics (fineness) of the recovered fly ashes, drying and processing to achieve properties for use in concrete were carried out. Initially, fly ash was oven dried at 105 ^oC and then screened at 600 µm. This material (referred to as ‘reference’) had a similar appearance to typical (dry) fly ash

and could flow. As noted above, the techniques used for processing were dry sieving, air classifying and grinding. The first two involve particle separation, with the fine fraction of these tested, while the latter reduced the size of all material considered. The target for processing was to achieve fly ash of fineness to Category S in BS EN 450-1 [17], i.e. not more than 12.0% retained on a 45 μm sieve, with suitable procedures established, for each of the above, to achieve this. Although means of assessing energy requirements were outwith the scope of the study, it should be recognised that these (and associated costs) would be different between the methods.

Dry sieving was used, since it represents a simple approach to separating material and developments have seen this receive attention for processing fly ash [6, 33]. The method is influenced by several factors [34], including the size and shape of particles relative to the sieve apertures and the mesh size itself. It has been noted [33] that with the differences in particle size distribution between fly ash and carbon particles, sieving can be effective in separating these. In the study, passage of material through a 63 μm sieve was carried out, which was suitable for the materials following drying and in achieving the above target (Category S) fineness.

Air classification is used with dry fly ash in practice [22, 35], so it meets fineness requirements for concrete and has been adopted in a few studies to process material [36, 37]. The method separates particles from an air stream (depending on their size and density) as they pass through a chamber of specific geometry, with the feed velocity normally controlling the size separation [35]. This was carried out for the study with an air classification unit by a company providing a separation service. Reductions in LOI with air classifying of fly ash have been noted to depend on the fineness of carbon [35].

Grinding provides a means of reducing particle size and is used in the cement industry, and its application with dry fly ash has also been reported in several studies [38-42]. Grinding of fly ash used a Fritsch Pulverisette laboratory ball mill, comprising two grinding bowls of 250 ml capacity, with 6 x 20 mm balls in each. The samples are primarily crushed by the impact of the grinding balls in the system [43] and this was carried out in batches for 20 minutes, at an average rotation speed. It was expected that the technique, given the closed system, would have little influence on the carbon content of fly ash.

6. Characteristics of Processed Materials

6.1 Physical properties and LOI

The physical properties and LOI of reference and processed fly ashes are shown in Table 4. Initial screening at 600 µm (i.e. reference material) lowered the 45 µm sieve retention by about 10 to 20%, with LOI reduced by about 0.5 to 1.0%. Similar values/slight increases in particle densities were also noted. The property ranking generally followed that of the recovered materials.

Sieving at 63 µm and air classifying effectively extended the changes with initial screening. In all but one case, material following these processes had 45 µm sieve retentions of 12.0% or less. The LOIs also reduced, with greatest effect for fly ashes with higher initial values and for 63 µm sieving (3.0 to 4.5%) compared to air classifying (0.5 to 2.0%). LOIs before and after processing using these techniques, in terms of BS EN 450-1 [17] categories, were as follows,

LFA1, Reference: Category B; 63 µm sieve: Category A; Air classifying: Category B
LFA2, Reference: non-conforming; 63 µm sieve: Category B; Air classifying: Category C
SFA1, Reference: non-conforming; 63 µm sieve: non-conforming; Air classifying: non-conforming

Increases in particle density of around 100 kg/m^3 were noted after 63 µm sieving, while similar/slightly greater values were found with air classifying, compared to the reference. The changes obtained correspond to the removal of coarser particles (fly ash and carbon), as well as those present in agglomerates, with agreements in ranking between LOI and particle density also noted. The differences in fly ash properties following separation are influenced by the feed (reference) material properties and factors associated with the processing methods, as described above.

Following grinding, all materials had increased fineness (0.6 to 8.1% retained on the 45 µm sieve). LOI was within about 1.0% of the reference, relating to the closed system. Particle density was found to increase for all materials and was higher for grinding than the other methods. This has been reported previously with dry fly ash [40] and is considered to reflect breakdown of (coarser) fly ash and porous carbon particles [38]. For wet material, this is also likely for agglomerates, while reductions in inter-particle space may also contribute.

The particle size distributions of the recovered and processed fly ashes are shown in Figure 1. These indicate some differences in ranking compared to 45 μm sieve retentions for the various materials. In general, after processing, grinding gave finest material, with air classification next, followed by 63 μm sieving. The greatest effects with the different processes tended to occur with increasing coarseness of the recovered fly ash.

Scanning electron microscopy images, showing the particle morphologies for LFA1 are given in Figure 2. Recovered material from this storage area comprised spherical particles (with a range of sizes) and some of irregular shape. There was also evidence of agglomeration and clustering of particles with roughening of their surfaces. These correspond to some of the effects referred to above, with features similar to those noted previously for wet-stored fly ash [e.g. 9, 10].

The images following 63 μm sieving and air classifying of fly ash resemble the recovered material, with agglomerates/clustering and greater quantities of smaller spherical particles, generally noted. The ground material indicates the presence of irregular/angular shaped particles and a few spheres. In this case, the effects are likely to correspond to the breakdown of coarser spherical fly ash particles, agglomerates present due to wet storage, as well as carbon.

6.2 Chemical Composition

The bulk oxide compositions obtained, using X-ray fluorescence spectrometry, are given for selective recovered and processed materials in Table 5. It is possible with 63 μm sieving and air classifying that differences occurring could correspond to reductions in carbon present [8]. Similarly, where components in fly ash concentrate in different size fractions, this may also be noted with these separation methods [37]. The closed system associated with grinding might be expected to have minor effects.

There were small variations in silica and alumina contents following 63 μm sieving and air classifying compared to recovered fly ash, with little influence of particle size. Similar differences were obtained between these components for recovered and ground materials. The presence of iron has been noted in fly ash particles greater than 20 μm [8], however, its contents didn't appear to be affected by processing. Calcium oxide was slightly higher in processed fly ash, with small differences in sulfate contents generally occurring. While potassium oxide changed little, reductions occurred with sodium oxide, which tended to follow chloride levels.

The main mineral components of selected fly ashes, estimated by Reitveld curve fitting, are given in Table 6. These show increases in crystalline/reductions in non-crystalline components with 63 μm sieving and air classifying, compared to recovered material. This differs from dry fly ash, where little change [44] or increases in crystalline phases (mainly mullite) have been found for coarser size fractions [45]. The reason for this is not known, however, for the values obtained, influences on the material's reactivity would be expected to be small. Greater changes were noted after grinding, with increases in crystalline/reductions in non-crystalline components. These may reflect exposure of internal (particle) crystalline material [42, 45], recorded during X-ray diffraction measurements. As (outer) fly ash particle surfaces remain accessible after grinding, the apparent reduction in non-crystalline (reactive) components would seem unlikely to influence reactivity.

7. Reaction Characteristics

The results from calorimetry tests evaluating heat development are shown in Table 7. These indicate small differences in time to main peak for reference and processed materials. The magnitude of peaks were comparable for the reference, 63 μm sieve and air classified fly ashes, with ground material higher. The total heat at 72 hours increased with processing and was also greatest for grinding. Given the limited reaction of fly ash during this early period, the results suggest that, as for dry material, with greater fineness following processing, the surface area may provide increased sites, thereby promoting PC hydration [23].

The calcium hydroxide contents using thermogravimetric analysis are given in Figure 3 for LFA1 and SFA1. The results follow typical behaviour for PC/fly ash, with initial increases reflecting PC hydration at early ages, and reductions with time due to pozzolanic reactions. The ground fly ashes tended to have higher calcium hydroxide contents at early ages, generally agreeing with the calorimetry tests, with little clear effect between the references and other processed fly ashes. Reductions in calcium hydroxide were noted from 7 to 14 days (later for the references), which is the period pozzolanic activity is typically noted from at standard curing temperatures [46]. These increased with processing of fly ash, compared to the references, with greatest effects at the end of the test by grinding.

8. Processed Material in Concrete

A concrete mix of average structural strength class was tested. This had a cement content of 350 kg/m^3, with fly ash at a level of 30%. The water content was 185 *l*/m^3 (with no admixtures) and the fine to total aggregate ratio 32%.

8.1 Fresh Properties

The results from consistence tests (slump) are shown against fly ash fineness (45 µm sieve retention) in Figure 4, and gave R^2 of 0.72. As indicated, slump was lowest for the reference fly ashes (70 to 115 mm), with increases as the materials became finer (and LOI reduced in some cases). In general, similar slump values were obtained for concrete containing the 63 µm sieved and air classified fly ashes, with greatest values following grinding. Overall, it was noted that a 10% reduction in 45 µm sieve retention gave an increase in slump of around 25 mm.

The benefits of fly ash (particularly finer material) on water demand of concrete are well known and relate to various effects including, particle shape and texture, dispersion of cement flocs and influences on particle packing [47, 48]. Wet storage has been found to reduce slump compared to dry fly ash concrete, which appears to reflect agglomeration/roughening of particles and their influences on factors affecting water demand [11]. The results with sieving and air classifying may relate to reduced coarse fly ash and carbon particles in the material and fewer agglomerates, i.e. removal of components tending to increase water demand, and agrees with the effects of air classifying on dry fly ash reported previously [36].

Research indicates that grinding of dry fly ash can cause water demand to both increase [39], and reduce (which may reflect fewer hollow particles present) and then increase [41], depending on the duration used. Factors including changes in surface area and particle shape of fly ash, and influences on particle packing have been noted to influence behaviour [39, 41, 42]. These would be expected to occur during the grinding of wet-stored fly ash, with breakdown of agglomerates also likely to affect the inter-particle space and shape of the material. It appears that for the grinding conditions used in the study, the various effects referred to give a net improvement in consistence of concrete.

8.2 Strength Development

The compressive (cube) strength development results for concrete are given in Figure 5, where a comparison between 28 day strength and fly ash fineness (45 μm sieve retention) is also made. The results follow typical strength development behaviour for fly ash concrete, with increases occurring during the 90 day test period, but at a reducing rate. Differences in strength of about 2.0 to 3.0 MPa were found between reference fly ash concretes to 28 days, with SFA1 tending to give higher strengths thereafter. Although, it is not possible to quantify the effects of wet storage on fly ash properties, referred to earlier, for example reactivity potential [11], these are likely to be reflected in the data.

The results also indicate small differences in strength between fly ash concretes at early ages, with benefits through processing tending to become more noticeable with time. The strength increases compared to the reference fly ashes ranged from 0.5 to 5.0 MPa for 63 μm sieve and air classified material from 28 days onwards, and 3.5 to 7.5 MPa for ground material over this period. A comparison of 28 day strength results and fineness for all materials (Figure 5(d)) shows general agreement between these, with gradual increases as 45 μm sieve retention reduced. Previous research has found similar behaviour with dry fly ash (unprocessed and processed) [40, 49, 50] and the results also reflect the thermogravimetric analysis described above.

The quantity of particles in dry fly ash less than 10 μm have been noted to influence the material's performance [36, 51]. To examine this for wet-stored processed fly ash, a comparison was made between sub 10 μm content and 28 day strength. For the reference fly ash concretes, the sub 10 μm contents of the recovered fly ashes were used (and in subsequent comparisons made for other concrete properties). The relationship, shown in Figure 6, indicates an R^2 of 0.81, suggesting that effects with dry fly ash may apply to wet material following processing. Furthermore, it appears that the sub 10 μm content, irrespective of the processing method, is an important factor affecting behaviour. Data in the current study indicate progressive increases in strength up to a sub 10 μm content of about 60% (ground fly ash).

8.3 Water Absorption and Intrinsic (Air) Permeability

Measurements of water absorption (initial surface absorption at 10 minutes (ISAT-10)) and intrinsic (air) permeability are shown against the sub 10 μm content of fly ash in Figure 7. Variability tests in the laboratory for these methods on comparable concretes, with a single operator, indicate typical coefficients of variation of

9.5% and 10.0% respectively. The data show similar type behaviour to that for strength, with reduced absorption and permeability with increasing sub 10 μm particle content of fly ash in concrete and R^2 values of 0.72 and 0.90 between these. The lowest water absorption and intrinsic (air) permeability results were obtained by grinding fly ash, with 63 μm sieved and air classified material giving less improvement, compared to the reference.

Studies investigating the effects of fly ash fineness in paste have noted that as this increases, the porosity and average pore size reduce [50]. Other work with fly ash in concrete at equal w/c ratio indicates slight reductions in absorption with increasing fineness and wet storage of the material [11]. It has also been suggested that the physical effect of fine grains of fly ash, densely packing in cement, reduce wall effects [52], enhancing the interfacial region between paste and aggregate, and thereby durability potential. The data suggest similar effects may occur with the processed wet-stored fly ash.

8.4 Durability

8.4.1 Carbonation

Accelerated carbonation test results at 12 weeks are given against fly ash sub 10 μm particle content in Figure 8. Previous work suggests that a week under the test conditions (See Table 3) for concrete, is equivalent to about 1.5 years of outside-protected exposure [53]. Variability tests with the method indicate a coefficient of variation of 9.0 % for similar concretes. The data give higher carbonation depths than found in earlier studies [49], however the reason for this is not known. They also show similarities to the properties above, with carbonation depth reducing with increasing fly ash sub 10 μm content in concrete. Thus, small improvements were achieved with 63 μm sieving and air classification, with greatest benefits by grinding.

Carbonation of concrete is influenced by the microstructure and pore fluid chemistry. A study investigating carbonation for dry and unprocessed wet-stored fly ash (at equal strength) gave similar results between these, with some differences in air transportation (permeability) rates and lime consumption also noted [54]. In the current study, water absorption and intrinsic (air) permeability reduce following processing (and increased fly ash fineness), with a comparable effect for calcium hydroxide (c.f. thermogravimetric analysis). Given the similar relationships between the water and air transport properties/carbonation and fly ash sub 10 μm content, it appears that physical influences have an important role in the process.

8.4.2 Chloride diffusion

Chloride diffusion results for the test concretes are given in Figure 9, with respect to the sub 10 µm content of fly ash. Typical variability data for the test with similar concretes give a coefficient of variation of around 5.0%. The results indicate low values for chloride diffusion, comparable to those obtained with the test method previously. Similar behaviour to that of the other properties considered above, with a gradual reduction in chloride diffusion with increasing fly ash sub 10 µm content in concrete, was also noted. Thus, the results for chloride diffusion, as with carbonation, indicate gradual improvements compared to the reference by 63 µm sieving and air classifying of fly ash, and greatest effects by grinding.

Chloride ingress is affected by the physical and chemical properties of concrete. The ability of fly ash to (i) densify the concrete microstructure and (ii) adsorb and chemically bind chloride, affect the process [55]. A study on dry fly ash concrete found increased resistance to chloride penetration with fineness (achieved by particle separation), reflecting influences on water demand (w/c ratio) and microstructure [56]. Other research suggests comparable chloride diffusion rates for dry and unprocessed wet-stored fly ash concrete (at equal strength) [54]. From the results in Section 8.3, it appears that, as with carbonation, the effects of processed wet-stored fly ash on the physical properties of concrete influence chloride diffusion. Further work would be necessary to establish the role of (ii) above, on the process.

9. Practical Implications

With the possible changes in sourcing of fly ash and the quantities available for recovery from wet-storage areas, there is growing interest in this material. The current study has examined potential for drying and processing wet fly ash for use in concrete. The materials sampled were relatively coarse and of medium to high LOI. It was possible by drying/initial screening and then sieving, air classifying, or grinding fly ash to achieve comparable handling properties to dry material and Category S fineness levels to BS EN 450-1 [17]. Although not the main focus, some reductions in LOI were obtained for the particle separation methods (sieving and air classifying) used.

The benefits achieved following drying and processing of wet fly ash were influenced by the degree to which its particle size was reduced, generally increasing as this became greater and appearing not to depend on the method used. The effects applied to the fresh and hardened properties of concrete, including strength, water

absorption and intrinsic (air) permeability, and aspects of durability, up to a sub 10 μm content of about 60% (with ground fly ash). Given the behaviour observed, greater enhancements may be possible with further reductions in particle size.

It is important to recognise that the energy (and cost) requirements between processing techniques will be different and what can be achieved for fly ash using a particular approach would, from a practical point of view, need to be balanced against these. This and factors including carbon removal, which may be necessary in some cases, scaling-up and other issues with wet-stored fly ash and its use in concrete are currently being investigated.

10. Conclusions

1. Relatively coarse fly ash samples of medium to high LOI were recovered from UK lagoon and stockpile storage areas. Following drying (105 ^{o}C) and initial screening (600 μm), processing was carried out by sieving (63 μm), air classifying and grinding. These enabled fineness, essentially of Category S to BS EN 450-1 [13] to be achieved (greatest with grinding). Sieving and air classifying also gave small reductions in LOI (particularly the former), with little or no change in this noted by grinding.

2. Small differences in the bulk chemistry of fly ash were generally found following processing compared to recovered material. However, some changes in mineral composition were obtained with processing, in particular grinding. Reactivity studies with paste indicated increases in heat development (at 72 hours) and reductions in calcium hydroxide following processing, compared to reference fly ashes. Greatest effects for these were found with grinding of the materials.

3. The consistence of concrete (slump) increased with fly ash fineness following processing, irrespective of the method used. Small differences between reference and processed fly ash concrete strengths were noted at early test ages, with benefits tending to become more noticeable with time. Concrete strengths at 28 days for reference and processed fly ashes could be related to the material's fineness (45 μm sieve retention) . The results also indicate an influence of fly ash sub 10 μm particle content on this, with improvements up to the 60% level obtained (for ground fly ash) during the tests.

4. Other concrete properties, including water absorption and intrinsic (air) permeability, carbonation and chloride diffusion could also be related to the sub 10 μm content of fly ash. Thus for the tests carried out, progressive improvements in performance were obtained with sieved and air classified material, compared to the reference, with grinding consistently giving best properties.

5. The practical implications of the study suggest that drying/screening of wet-stored fly ash to re-establish handling properties and the application of processes to achieve finer fractions/reduce particle size, provide options for its recovery and use as an addition in concrete. Other issues associated with processing and its effects require to be investigated and are being considered in a related study.

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List of Tables

Table 1. Summary of test methods used for characterizing fly ash in the study

Property	**Relevant Method/Standard**	**Specimen Details**	**Test Arrangement**
Fineness/Particle Size Distribution	BS EN 451-2 [19]	1.0 g powder sample/ 45 μm sieve	Wet-sieving carried out for 1 minute under fixed pressure. Residue after drying compared to original sample mass.
	Laser particle size analysis+	1.0 g dispersed in 800 ml of water in ultrasonic bath	Particle size distribution determined from degree of scattering of collimated laser beam passing through the sample and using equipment software.
Loss-On-Ignition (LOI)	BS EN 196-2 [20]	1.0 g powder sample in a crucible	Ignition of sample carried out at 950 ^{o}C for 1 hour. Change in mass compared to initial sample mass.
Particle Density	BS EN 196-6 [21]	5.0 g sample in de-ionised/de-aired water	Pyknometer (50 ml) with fly ash sample vacuum de-aired. Particle density determined by displacement of water.
Bulk Oxide Composition	X-ray fluorescence+	Pressed homogenised powder pellet	X-ray fluorescence spectrometer with Rh Kα source.
Mineralogical Composition	X-ray diffraction+	Homogenised powder sample	X-ray diffractometer with Cu Kα source. Internal standard (5.0% corundum) and Rietveld curve fitting used for analysis.
Morphology	Scanning electron microscopy+	Fly ash layer on carbon tape/aluminium stub, gold/palladium coated	Scanning electron microscope with 15 kV accelerating voltage.

Note: + Tests following in-house methods as indicated

Table 2 Physical and chemical characteristics of PC and recovered fly ash samples

Property	PC	Fly Ash Samples		
		Lagoon Ash		Stockpile Ash
		LFA1	LFA2	SFA1
Fineness, 45µm sieve retention, %	364*	48.2	44.0	33.8
LOI, %	1.4	6.4	10.3	15.1
Particle Density, kg/m^3	3140	2120	1970	1980
Particle Size Distribution, µm				
d_{10}	-	6.2	4.6	2.6
d_{50}	-	46.1	40.6	21.9
d_{90}	-	133.6	139.0	95.7
Bulk Oxide Composition, % by mass				
SiO_2	21.5	48.3	46.9	49.8
Al_2O_3	5.4	27.2	28.7	23.9
Fe_2O_3	2.6	6.2	5.1	5.7
CaO	64.2	2.9	1.8	2.4
MgO	2.6	1.6	1.7	1.2
TiO_2	0.3	1.5	1.4	1.4
K_2O	0.7	1.2	1.5	2.1
Na_2O	0.3	1.4	0.8	0.5
P_2O_5	0.1	0.6	0.4	0.8
Cl	0.0	2.1	1.0	0.0
SO_3	2.8	0.5	0.5	0.6
Main Mineral Composition, % by mass				
Quartz	-	10.8	4.6	3.8
Mullite	-	16.6	18.0	8.0
Non-crystalline+	-	65.3	66.8	72.7

* Specific surface area, m^2/kg (Blaine method)

\+ By difference

Table 3. Summary of test methods used for paste and concrete in the study

Material / Property	**Relevant Method/Standard**	**Specimen Details**	**Test Arrangement**
Paste			
Heat Development	Iso-thermal Conduction calorimetry+	PC/30% fly ash freshly mixed paste (w/c ratio 0.5)	Test carried out for heat development over 72 hours. Electrical output from equipment monitored to enable heat evolution rates to be determined.
Hydration/Pozzolanic Activity	Thermogravimetric analysis+	Powder sample from PC/30% fly ash paste (w/c ratio 0.5) 25 mm cube (over-dried at 105 ^{o}C).	$Ca(OH)_2$ determined by mass change in N_2 atmosphere over temperature range 450 to 510 ^{o}C during a rise to 1000 ^{o}C at 20 ^{o}C/min. Tests carried out on samples up to 90 days.
Concrete			
Consistence (Slump)	BS EN 12350–2 [27]	300 mm cone of hand-compacted concrete	Reduction in height on removal of cone taken as measure of consistence.
Compressive Strength	BS EN 12390–3 [28]	2 No. 100 mm cubes	Tested in compression periodically up to 90 days.
Water Absorption	Initial surface absorption BS 1881–208 [29]	2 No. faces 150 mm cube (oven-dried at 105 °C)	200 mm head of water applied for 10 minutes. Measurement of rate of water absorption into specimen through capillary tube taken at this time.
Intrinsic (Air) Permeability	Dundee University [30]	54 mm φ × 50 mm cores taken from 100 mm cubes (oven-dried at 105 °C)	Specimens positioned in permeability cell. Air pressures from 0.21 to 0.76 MPa applied until steady-state achieved. Calculation based on Darcy's Law.
Carbonation	BS 1881–210 [31]	75 mm × 75 mm × 300 mm prism (dried in laboratory air for 14 days, then wax sealed on 5 faces)	Specimens exposed to 4.0% CO_2, 20 °C, 55% relative humidity for 12 weeks. Carbonation depth measured on slice removed from prism, using pH indicator.
Chloride Diffusion	Dundee University [32]	100 mm φ × 25 mm slices taken from 300 mm cylinder (24 hour pre-saturation)	Specimens in diffusion cells, downstream $Ca(OH)_2$/partially immersed in 5.0 M NaCl solution. Calculation of diffusion coefficient based on Fick's First Law.

Note: + Tests following in-house methods as indicated

Table 4 Physical characteristics and LOI of reference and processed fly ashes

Samples		Characteristics of Fly Ash		
		Fineness, 45 µm Sieve Retention, %	**LOI, %**	**Particle Density, kg/m^3**
LFA1	Reference*	26.8	6.1	2120
	63 µm sieved	12.0	3.2	2210
	Air classified	6.1	5.8	2120
	Ground	8.1	5.3	2310
LFA2	Reference	33.4	9.7	2010
	63 µm sieved	8.7	5.1	2130
	Air classified	8.1	7.6	2020
	Ground	6.3	10.8	2170
SFA1	Reference	23.8	14.2	2060
	63 µm sieved	10.2	10.7	2160
	Air classified	12.9	13.1	2100
	Ground	0.6	14.1	2230

* Recovered material screened at 600 µm

Table 5 Bulk oxide composition of selective recovered and processed fly ashes, % by mass

Samples	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	K_2O	Na_2O	P_2O_5	Cl	SO_3
LFA1											
Recovered	48.3	27.2	6.2	2.9	1.6	1.5	1.2	1.4	0.6	2.1	0.5
63 µm sieved	48.3	27.6	7.7	3.7	2.1	1.5	1.3	0.5	0.6	0.5	1.1
Air classified	47.6	29.9	6.1	3.4	2.3	1.6	1.2	0.7	0.8	0.6	0.7
Ground	50.0	27.1	5.7	3.3	1.9	1.4	1.3	0.6	0.5	0.8	0.6
LFA2											
Recovered	46.9	28.7	5.1	1.8	1.7	1.4	1.5	0.8	0.4	1.0	0.5
Air classified	44.7	26.8	5.9	5.9	1.7	1.4	1.6	0.5	0.4	0.0	0.7
Ground	49.0	26.9	6.7	2.4	1.9	1.4	1.5	0.3	0.4	0.1	0.4

Table 6 Main mineral composition of selective recovered and processed fly ashes, % by mass

Samples		Quartz	Mullite	LOI	Non-crystalline*
LFA1	Recovered	10.8	16.6	6.4	65.3
	63 μm sieved	10.9	22.3	3.2	60.9
	Air classified	8.7	26.3	5.8	57.3
	Ground	16.8	30.4	5.3	45.7
LFA2	Recovered	4.6	18.0	10.3	66.8
	Ground	10.7	32.6	10.8	43.7
SFA1	Recovered	3.8	8.0	15.1	72.7
	Air classified	5.3	12.6	13.1	68.2

* By difference

Table 7 Heat evolution characteristics of reference and processed fly ashes

Samples		Peak Rate		Total Heat, kJ/kg (72 hours)
		Time, hours	Value, W/kg	
LFA1	Reference*	9.0	2.97	204.0
	63 µm sieved	8.6	2.98	208.4
	Air classified	8.0	3.06	206.5
	Ground	9.3	3.20	222.4
SFA1	Reference	9.0	2.98	204.0
	63 µm sieved	9.6	2.90	207.7
	Air classified	8.7	3.00	209.7
	Ground	8.6	3.23	217.3

* Recovered material screened at 600 µm

List of Figures

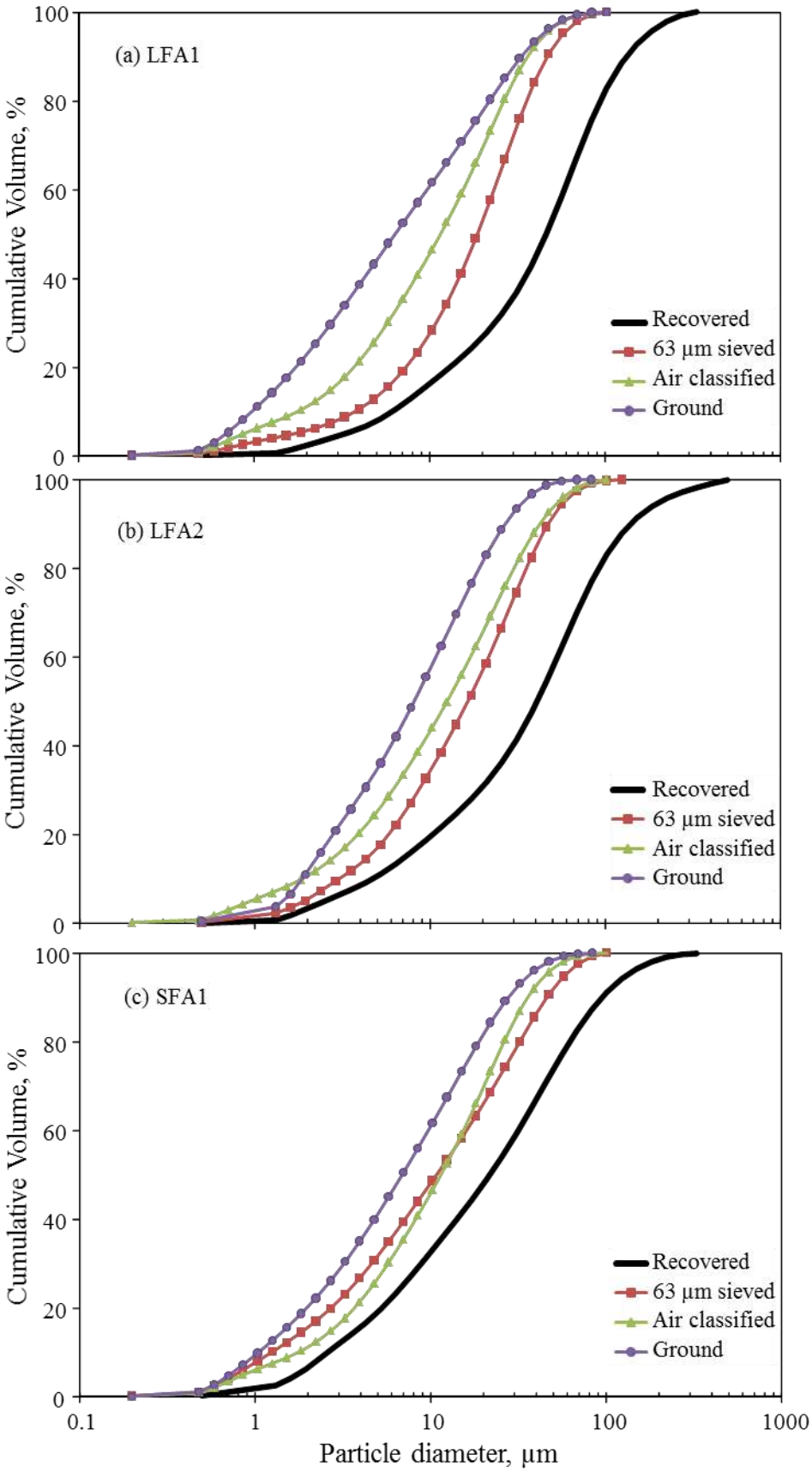
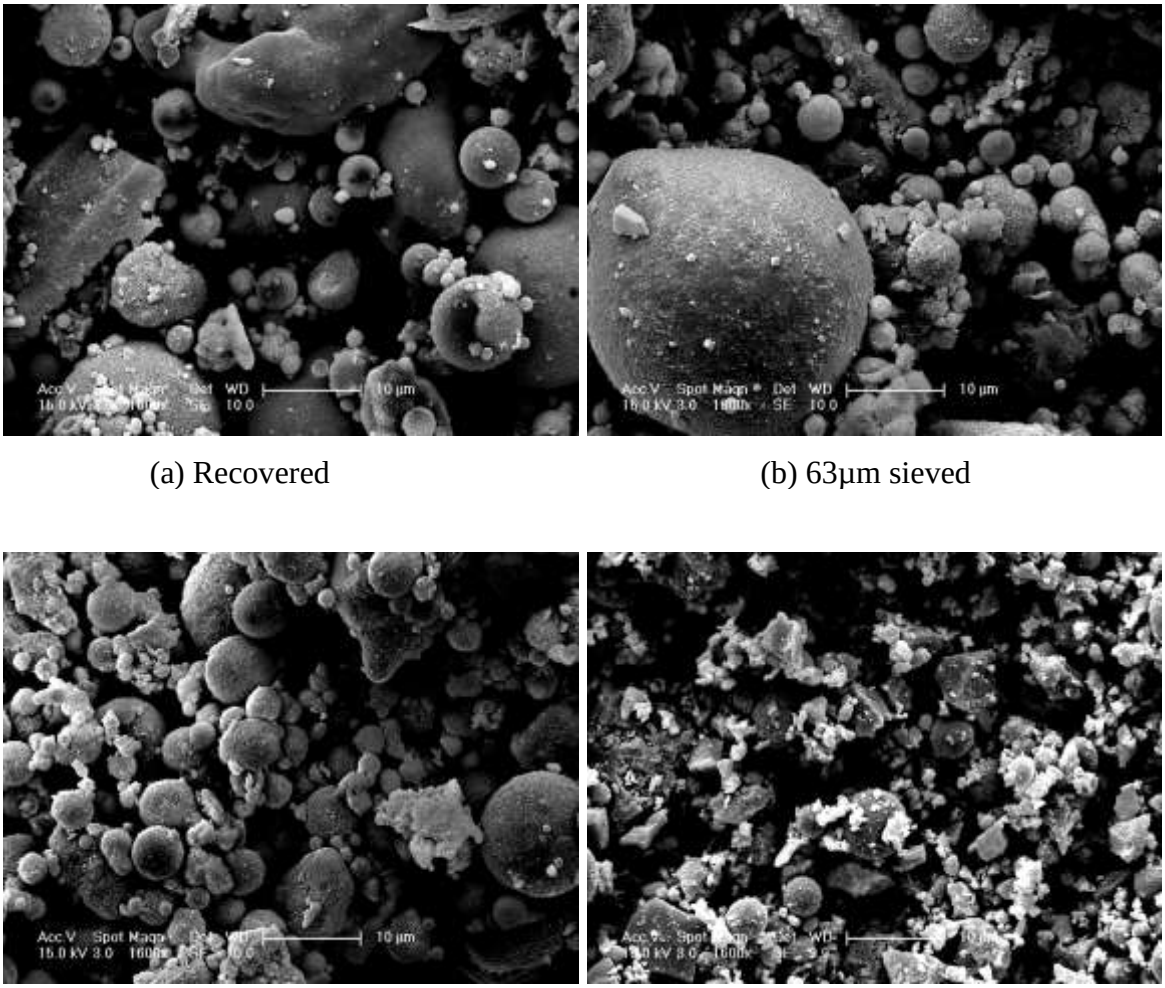


Figure 1 Typical particle size distributions of recovered and processed fly ashes for (a) LFA1, (b) LFA2, (c) SFA1



(a) Recovered (b) 63µm sieved

(c) Air classified (d) Ground

Figure 2 (a) to (d) Scanning electron microscopy images of recovered and processed LFA1 (1600x)

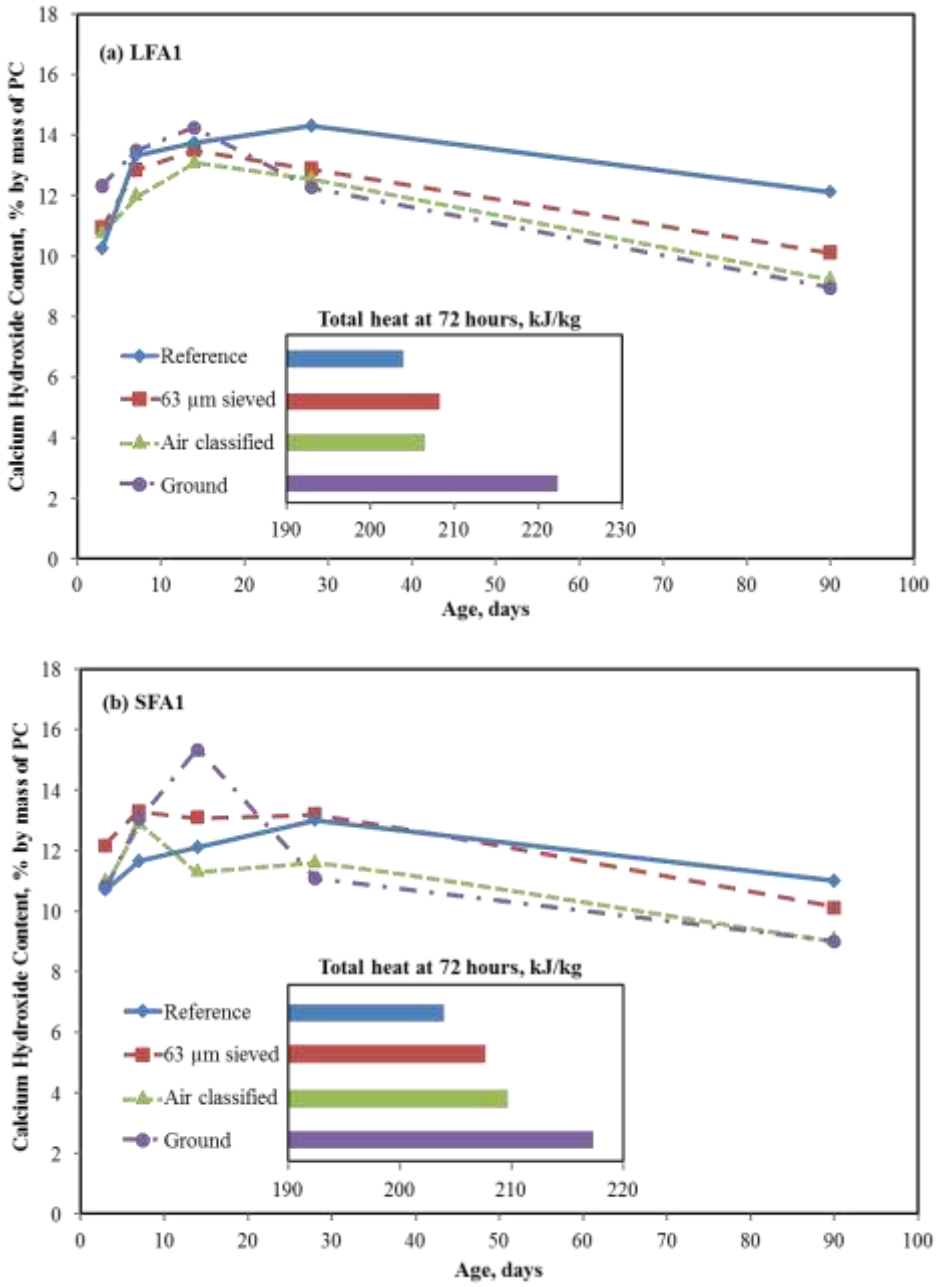


Figure 3 Change in calcium hydroxide of paste (w/c = 0.50) with time for reference and processed (a) LFA1 and (b) SFA1

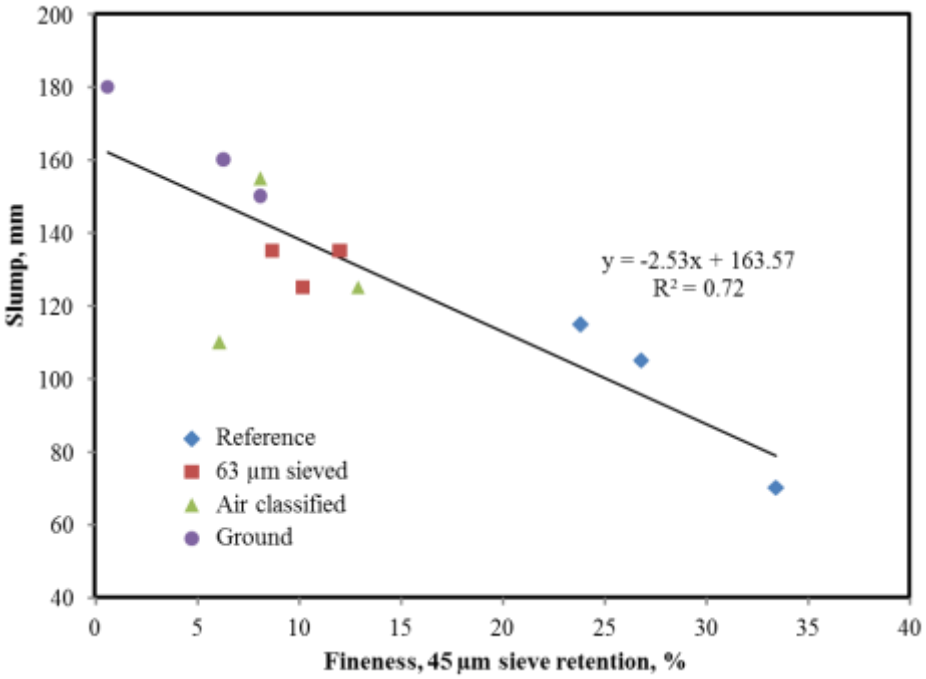


Figure 4 Effect of fly ash fineness (45 µm sieve retention) on slump of concrete (w/c = 0.53)

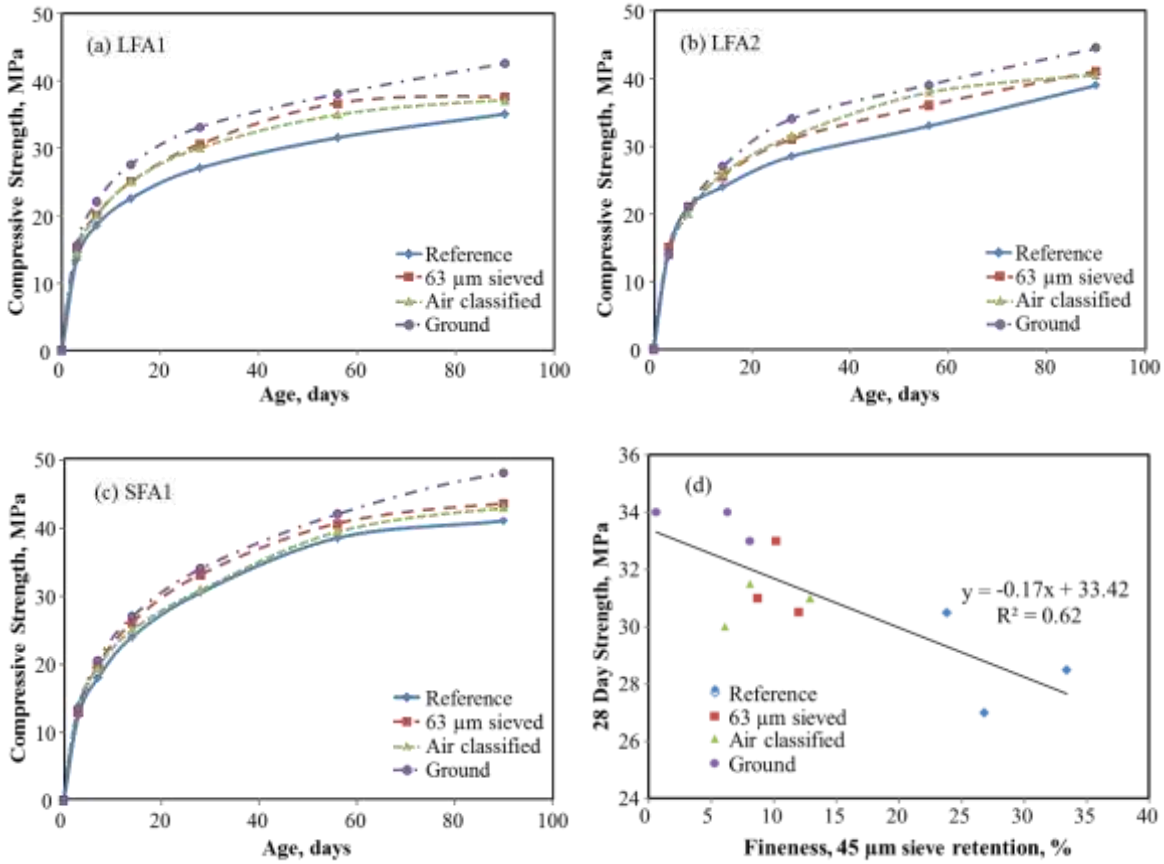


Figure 5 (a) to (c) Strength development of fly ash concretes (w/c = 0.53) and (d) relationship between fly ash fineness (45 µm sieve retention) and 28 day compressive strength (cube)

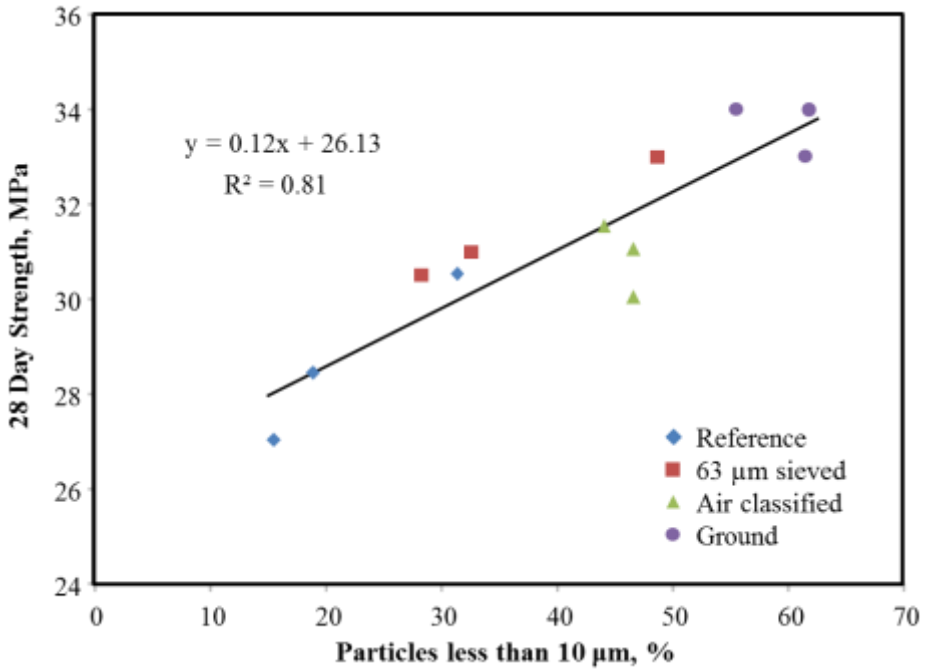


Figure 6 Relationship between sub 10 µm content of fly ash and 28 day compressive strength (cube) of concrete (w/c = 0.53)

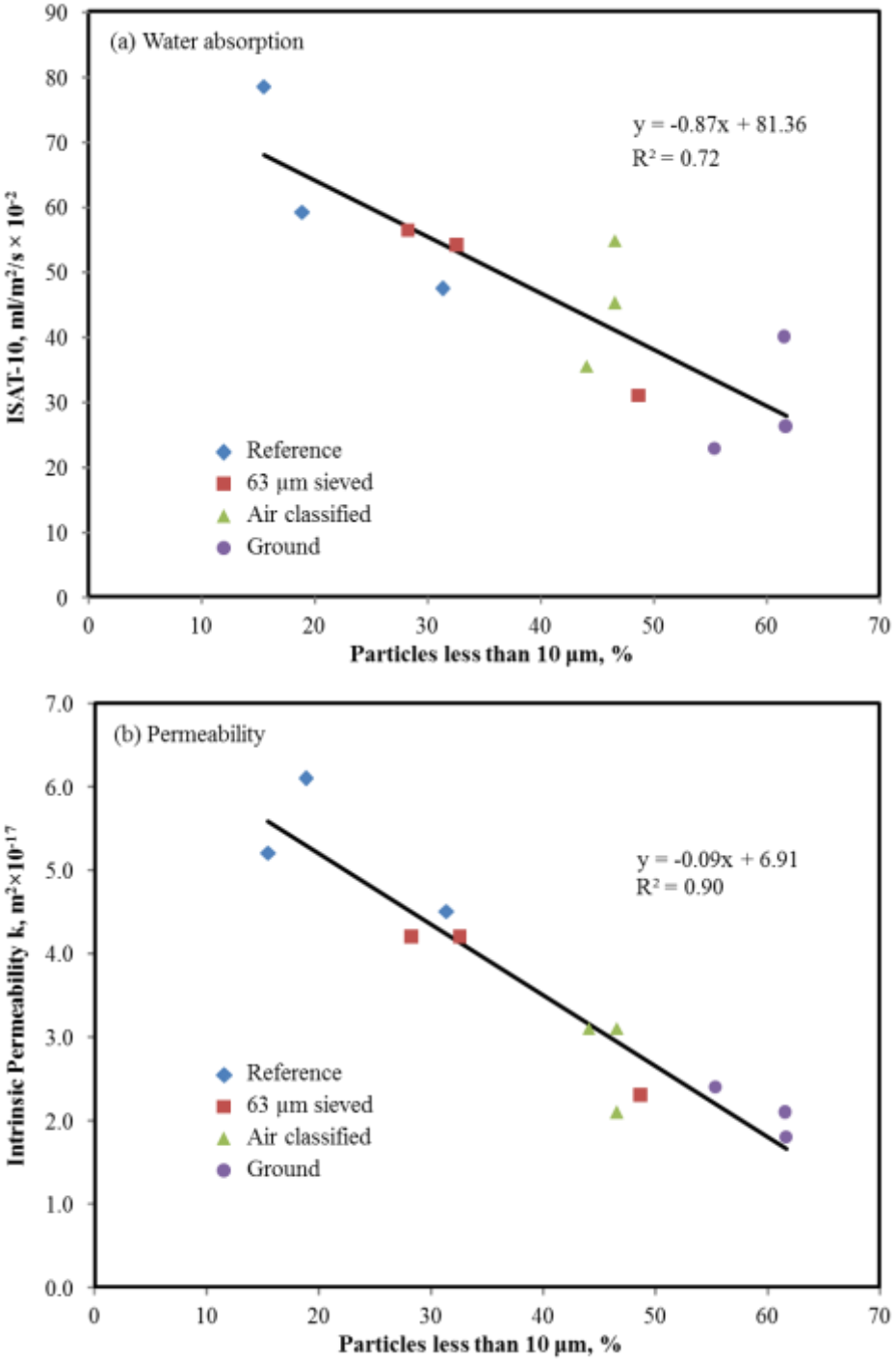


Figure 7 Relationship between sub 10 µm content of fly ash and (a) water absorption (ISAT-10) and (b) permeability (intrinsic (air)) of concrete (w/c = 0.53)

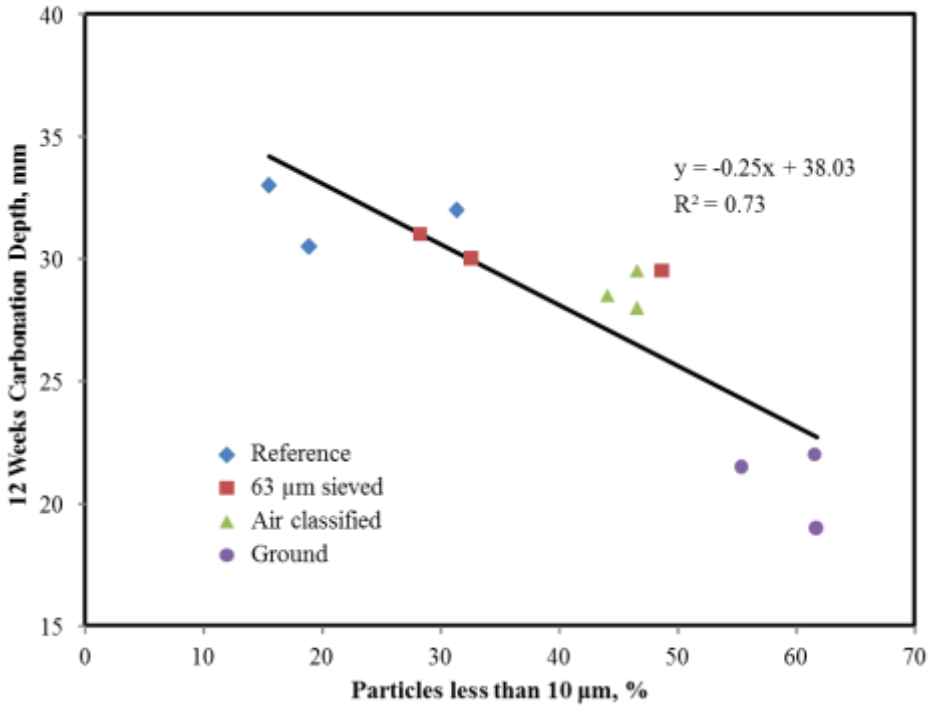


Figure 8 Relationship between sub 10 µm content of fly ash and carbonation depth of concrete (w/c = 0.53; Exposure: 20 °C, 4.0% CO_2, 55% relative humidity)

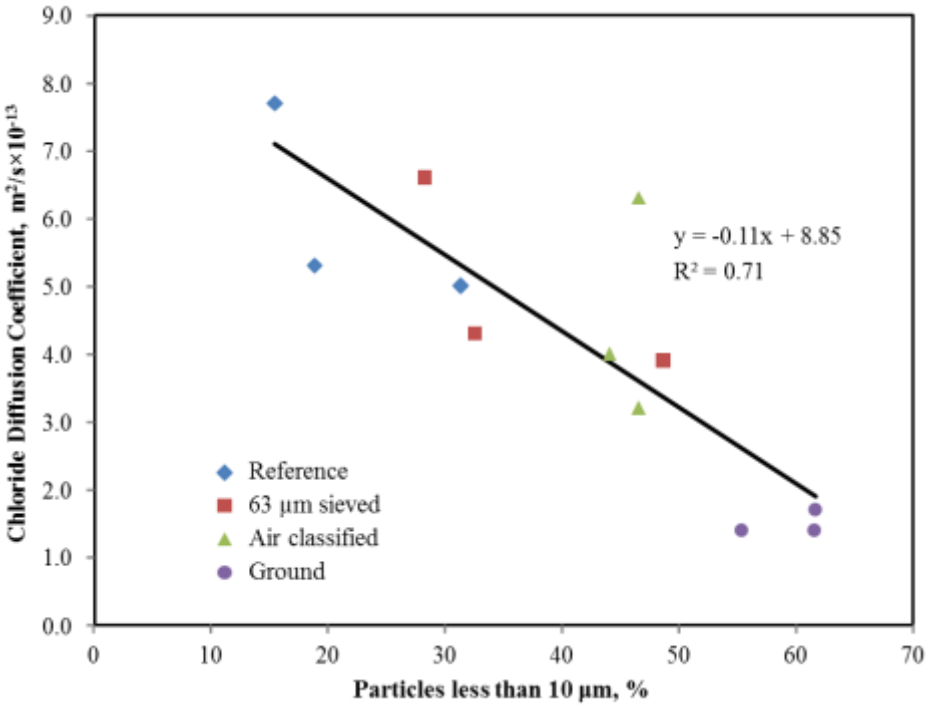


Figure 9 Relationship between sub 10 µm content of fly ashes and chloride diffusion coefficient of concrete (Two compartment cell, 20 °C, upstream 5.0 M NaCl/downstream saturated $Ca(OH)_2$)